Resolving the Benzo(j)fluoranthene Challenge

Separate New PAHs Quickly Using the Rxi™-17 GC Column

By Robert Freeman, Environmental Innovations Chemist

- Fully resolve benzo(j)fluoranthene from benzo(b) & (k).
- Excellent resolution of 16 priority pollutant PAHs.
- Separate difficult dibenzo pyrene isomers.

New Compounds, New Challenges

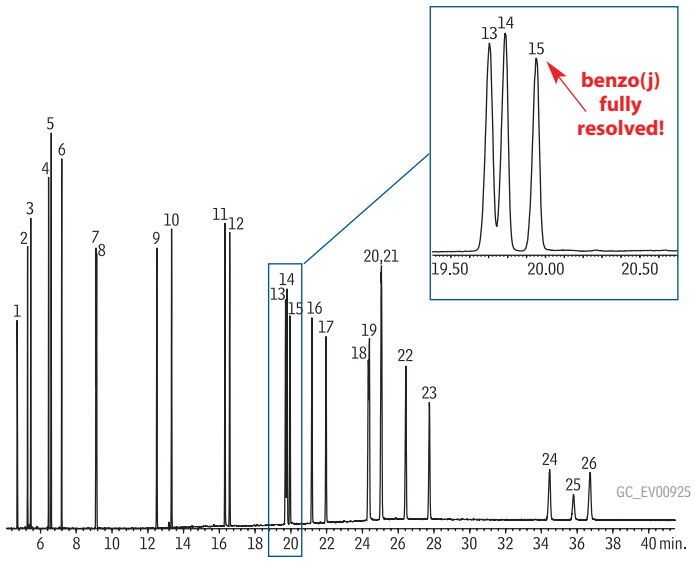
Polynuclear aromatic hydrocarbons (PAHs) are widespread organic pollutants that significantly affect environmental quality and raise human health concerns. The US EPA mandates testing of 16 priority PAH pollutants, while analyte lists in other countries are expanding to include compounds such as benzo(j)fluoranthene, dibenzo(a,h)-acridine, and dibenzo(a,e)pyrene, that are difficult to analyze under conventional test conditions. Benzo(j)fluoranthene and benzo(b)fluoranthene, for example, co-elute on a 5%diphenyl/95%dimethyl polysiloxane stationary phase. When reporting of individual concentrations for each isomer is required, conventional methods are not viable and new solutions must be found.

The Rxi™ Alternative

The Rxi™-17 column contains a 50% diphenyl/50% dimethyl polysiloxane stationary phase. The higher concentration of phenyl groups in this stationary phase increases retention of phenyl-containing compounds, such as PAHs, thus facilitating separation. We also used a Drilled Uniliner® inlet liner since it eliminates sample exposure to cold spots and potentially active metal components in the injection port. Using a pulsed splitless injection, we maximize sample transfer to the column while minimizing high molecular weight discrimination.

The data in Figure 1 demonstrate the excellent resolution of benzo(j)fluoranthene achievable on the Rxi™-17 column. Phenanthrene and anthracene also resolve well on this column under slower run conditions (data not shown). Using the Rxi™-17 column with an optimized temperature program is a practical solution to the challenges posed by expanding PAH analyte lists. If you are struggling to quantify PAHs on conventional columns, try the Rxi™-17 column and the optimized temperature program shown here.

Figure 1 Fast, effective separation of target PAHs using an Rxi™-17 column and an optimized temperature program.



Peak List	Ret. Time (min.)		Ret. Time (min.)
1. naphthalene	4.70	14. benzo(k)fluoranthene	19.78
2. 1-methylnaphthalene	5.28	15. benzo(j)fluoranthene	19.95
3. 2-methylnaphthalene	5.46	16. benzo(a)pyrene	21.17
4. acenaphthylene	6.45	17. 3-methylcholanthrene	21.97
5. acenaphthene	6.60	18. dibenzo(a,h)acridine	24.33
6. fluorene	7.18	19. dibenzo(a,j)acridine	24.39
7. phenanthrene	9.10	20. indeno(1,2,3-cd)pyrene	25.04
8. anthracene	9.14	21. dibenzo(a,h)anthracene	25.07
9. fluoranthene	12.50	22. benzo(ghi)perylene	26.43
10. pyrene	13.33	23. 7H-dibenzo(c,g)carbazole	27.75
11. benzo(a)anthracene	16.32	24. dibenzo(a,e)pyrene	34.46
12. chrysene	16.58	25. dibenzo(a,i)pyrene	35.80
13. benzo(b)fluoranthene	19.70	26. dibenzo(a,h)pyrene	36.73

Column: Rxi™-17, 30m, 0.25mm ID, 0.25µm (cat.# 13523)
Sample: PAH mix, 20µg/mL each component: EPA Method 610 Mix (cat.# 31011), PAH Supplement Mix (cat.# 31857), 1-methylnaphthalene (cat#31283), 2-methylnaphthalene (cat#31285); Inj.: 1.0µL pulsed splitless injection (20ng each component on column), 4mm Drilled Uniliner® inlet liner with hole at top (cat # 21055); pulse: 20psi @ 0.3 min., 40mL/min. @ 0.2 min. Inj. temp.: 300°C; Carrier gas: helium, constant flow; Flow rate: 1.2mL/min.; Oven temp.: 90°C (hold 1.0 min.) to 215°C @ 25°C/min. (hold 0.5 min.) to 235°C @ 4°C/min., to 280°C @ 15°C/min., to 320°C @ 4°C/min. (hold 20 min.); Det.: Agilent 5973 GC/MS; Scan range: 50-550 amu; Solvent delay: 4.0 min.; Tune: DFTPP; Ionization: EI

Rxi™-17 Columns (fused silica)

(Crossbond® 50% diphenyl / 50% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.25mm	0.25	40 to 280/300°C	30-Meter	13523

Direct Injection Liners for Agilent GCs

ID* x OD & Length (mm)	qty.	cat.#
Drilled Uniliner® (hole on top)		
4.0 ID x 6.3 OD x 78.5	5-pk.	21055

SV Calibration Mix #5 / 610 PAH Mix (16 components)

acenaphthene
acenaphthylene
anthracene
benzo(a)anthracene
benzo(a)pyrene
benzo(b)fluoranthene
benzo(k)fluoranthene
benzo(ghi)perylene
chrysene
dibenzo(a,h)anthracene
fluoranthene
fluorene
indeno(1,2,3-cd)pyrene
naphthalene
phenanthrene
pyrene

2,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31011

PAH Supplement Mix for Method 8100 (8 components)

benzo(j)fluoranthene
dibenzo(a,h)acridine
dibenzo(a,j)acridine
7H-dibenzo(c,g)carbazole
dibenzo(a,e)pyrene
dibenzo(a,h)pyrene
dibenzo(a,i)pyrene
3-methylcholanthrene

1000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31857

HROMalytic RESTEK '07
Australian Distributors ECHnology
Tel: 03 9762 2034 Fax: 03 9761 1169 www.chromtech.net.au info@chromtech.net.au